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PREPARATION OF RIGID POLYURETHANE FOAMS USING MIXED METAL COMPLEXES  
AND TETRAETHYLENEPENTAMINE AS CATALYSTS

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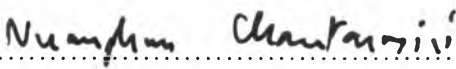
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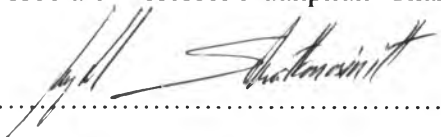
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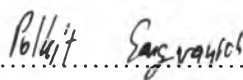
  
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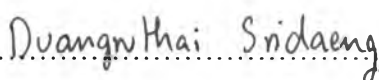
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มานิตา เรื่องศรี : การเตรียมโฟมพอลิยูรีเทนแบบแข็งโดยใช้สารประกอบเชิงซ้อนของโลหะผสมและเทตระเอทิลีนเพนทามีนเป็นตัวเร่งปฏิกิริยา. (PREPARATION OF RIGID POLYURETHANE FOAMS USING MIXED METAL COMPLEXES AND TETRAETHYLENEPENTAMINE AS CATALYSTS) อาจารย์ที่ปรึกษาวิทยานิพนธ์หลัก: รศ.ดร. นवलพรรณ จันทร์ศิริ. 122 หน้า.

งานวิจัยนี้ได้พัฒนาตัวเร่งปฏิกิริยาสำหรับเตรียม โฟมพอลิยูรีเทนแบบแข็ง โดยใช้ตัวเร่งปฏิกิริยาเป็นสารประกอบเชิงซ้อนของโลหะ-แอมีน  $[M_1(\text{tetraen})]$  ซึ่ง  $M_1 = \text{Cu, Zn, Ni, Co}$  และ  $\text{Mn}$  และสารประกอบเชิงซ้อนของโลหะผสม-แอมีน  $[M_2(\text{tetraen}):M_1(\text{tetraen})]$  ซึ่ง  $M_2 = \text{Cu}$  และ  $M_1 = \text{Zn, Ni, Co}$  และ  $\text{Mn}$  พิสูจน์เอกลักษณ์ของตัวเร่งปฏิกิริยาด้วยเทคนิคอินฟราเรดสเปกโทรสโกปี ยูวี-วิสสิเบิลสเปกโทรสโกปี แมสสเปกโทรเมทรี และวิเคราะห์หาธาตุองค์ประกอบ สำหรับการเตรียมโฟมพอลิยูรีเทนแบบแข็งได้ศึกษาเวลาที่ใช้ในการทำปฏิกิริยา สมบัติทางกายภาพ และสมบัติเชิงกล โดยเปรียบเทียบผลการศึกษากับตัวเร่งปฏิกิริยาทางการค้า คือ ไดเมทิลไซโคลเฮกซิลแอมีน (DMCHA) ใช้เทคนิคเอทีอาร์-อินฟราเรดสเปกโทรสโกปีเพื่อพิสูจน์เอกลักษณ์ของพอลิยูรีเทน โฟมแบบแข็งและศึกษาการเปลี่ยนแปลงของหมู่ไอโซไซยาเนต (NCO conversion) การเตรียมโฟมพอลิยูรีเทนแบบแข็งจากตัวเร่งปฏิกิริยาสารประกอบเชิงซ้อนของโลหะผสม-แอมีน  $\text{Cu}(\text{tetraen}):Zn(\text{tetraen})$  ให้ความเวลาในการทำปฏิกิริยา ความสูงของโฟม และความหนาแน่นที่ดี ความหนาแน่นและความสามารถทนทานต่อแรงกดอัด (compressive strength) ของโฟมพอลิยูรีเทนแบบแข็งที่เตรียมจาก  $\text{Cu}(\text{tetraen}):Zn(\text{tetraen})$  ที่ดัชนีไอโซไซยาเนต 150 เท่ากับ  $49.8 \text{ kg/m}^3$  และ  $341.9 \text{ kPa}$  ตามลำดับ ซึ่งมีค่ามากกว่าความหนาแน่นและความสามารถทนทานต่อแรงกดอัดของโฟมที่เตรียมจากตัวเร่งปฏิกิริยาทางการค้าที่ดัชนีไอโซไซยาเนต 150 ซึ่งมีค่าเท่ากับ  $45.8 \text{ kg/m}^3$  และ  $309.7 \text{ kPa}$  ตามลำดับ

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MANITA RUANGSRI: PREPARATION OF RIGID POLYURETHANE  
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PENTAMINE AS CATALYSTS. ADVISOR: ASSOC. PROF. NUANPHUN  
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In this research, the catalysts for rigid polyurethane foams were developed by using metal-amine complexes [ $M_1(\text{tetraen})$ ] (where;  $M_1 = \text{Cu, Zn, Ni, Co}$  and  $\text{Mn}$ ) and mixed metal-amine complexes [ $M_2(\text{tetraen}):M_3(\text{tetraen})$ ] (where;  $M_2 = \text{Cu, } M_3 = \text{Zn, Ni, Co}$  and  $\text{Mn}$ ). The metal-amine and mixed metal-amine complexes were characterized by infrared spectroscopy, UV-visible spectroscopy, mass spectrometry and elemental analysis. Reaction times, physical and chemical properties of the prepared foams were investigated and compared with those prepared using commercial catalyst, dimethylcyclohexylamine (DMCHA). ATR-FTIR spectroscopy was used to characterize rigid polyurethane foams and determine isocyanate conversion. Rigid polyurethane foams catalyzed by mixed metal-amine complex of  $\text{Cu}(\text{tetraen}):Zn(\text{tetraen})$  had good reaction times, volume and density. Density and compressive strength of the foams at NCO index of 150 catalyzed by  $\text{Cu}(\text{tetraen}):Zn(\text{tetraen})$  were  $49.8 \text{ kg/m}^3$  and  $341.9 \text{ kPa}$ , respectively. They showed higher density and compressive strength than the foams catalyzed by DMCHA which were  $45.8 \text{ kg/m}^3$  and  $309.7 \text{ kPa}$ , respectively.

Field of Study: Petrochemistry and Polymer Science Student's Signature Manita Ruangsri

Academic Year: 2011 Advisor's Signature Nuanphun Chantarasi

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## LIST OF ABBREVIATIONS

%	percentage
$\epsilon$	Molar absorptivity
cm	centimeter
$\text{cm}^{-1}$	unit of wavenumber
$^{\circ}\text{C}$	degree Celsius (centigrade)
Cu(tetraen)	copper-tetraethylenepentamine complex
Cu(tetraen):Zn(tetraen)	copper-tetraethylenepentamine and zinc – tetraethylenepentamine complex
ATR-FTIR	Attenuated Total Reflectance Fourier Transform Infrared Spectrophotometer
DBTDL	dibutyltin dilaurate
DMCHA	N,N-dimethylcyclohexylamine
EA	Elemental Analysis
en	ethylenediamine
FTIR	Fourier Transform Infrared Spectrophotometer
g	gram
h	hour
HDI	Hexamethylene Diisocyanate
IDT	Initial Decomposition Temperature
ATR-IR	Attenuated- Infrared Total Reflectance
kg	kilogram
kV	kilovolt
M(OAc) <sub>2</sub>	metal acetate
m <sup>3</sup>	cubic meter
MDI	4,4'-methane diphenyl diisocyanate
mg	milligram
min	minute
mL	milliliter
mmol	millimole
NCO	isocyanate

OHV	hydroxyl value
pbw	part by weight
PIR	polyisocyanurate
PMDI	polymeric 4,4'-methane diphenyl diisocyanate
PUR	polyurethane
rpm	round per minute
RPUR	rigid polyurethane
RT	room temperature
s	second
SEM	Scanning Electron Microscope
S.D.	standard deviation
t	time
TDI	Toluene Diisocyanate
TGA	thermogravimetric analysis
$T_{\max}$	maximum core temperature
tetraen	tetraethylenepentamine
trien	triethylenetetramine
UV	ultraviolet spectroscopy
Zn(tetraen)	zinc-tetraethylenepentamine complex